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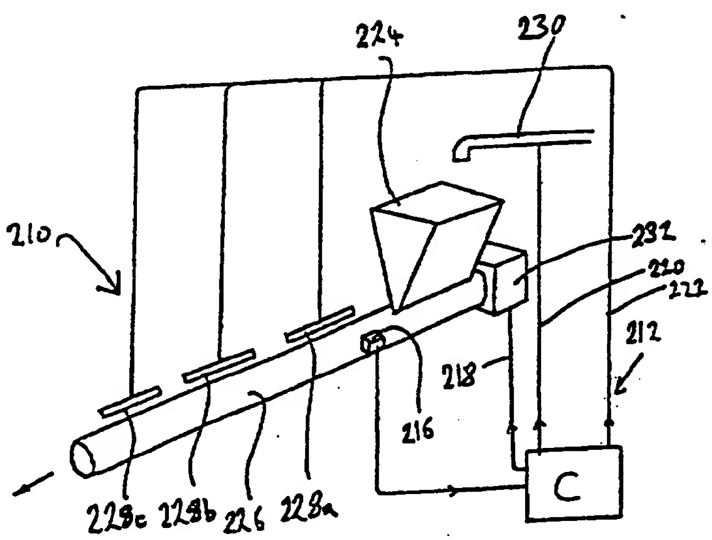

(57) Abstract

A chemical reactor apparatus (210) especially, but not exclusively in relation to producing a flow of polymers comprises test means (240) adapted in use to test a reaction product, or for testing an intermediate reaction product, reaction condition means (216, 252) adapted in use to detect one or more reaction conditions, and control means (212) adapted in use to control at least one reaction input parameter, in which in use the reaction condition means (216, 252) provides to the control means (212) a signal indicative of at least one reaction condition, and the test means (240) provides the control means (212) with a second signal indicative of a property of the reaction product being produced, and in which the control means (212) utilises both the first and the second signals in controlling the reaction input parameter.

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REACTION APPARATUS AND CONTROL SYSTEMS THEREFOR

This invention relates to reaction apparatus, for example apparatus for reacting constituents such as isomers with initiators and stabilisers to produce a polymer, and also to control systems for them. It may be especially (but not exclusively) useful in relation to apparatus for producing a flow of polymer material, and also to control systems for such apparatus.

It will be helpful to put the present invention into perspective by considering prior art systems for producing polymers.

A first prior art way of producing a polymer material, for example a plastics material polymer suitable for making plastics articles, is a small scale, one-off, batch method. A known quantity of a first start material, and possibly of a second start material, are put in a known reactor vessel, with a known amount of reaction initiator (if required) and reacted at a known temperature for a known time. The reaction product is then removed (and usually used to make something). This method has very little control system: all of the reaction parameters are predetermined beforehand and the system set to the required conditions. There may be a monitoring of temperature and/or stirring speed, and feedback control to ensure that they are as desired. This method produces batches of material. It cannot be changed to produce larger batches without first determining what are the new amounts of reagent, and their proportions, and new temperature (if different), time of "working" and mixing rate control. This is done on an individual

batch basis from manual look-up tables, or by trial and error.

A second prior art method of producing a plastics polymer material is to have a continuous process in which start materials are continuously, or periodically, introduced at a start end of a reaction plant, are reacted or processed, and emerge continuously, or periodically, from another part of the reaction plant. The control system for such "through-flow" processes is one of two known kinds.

A first control process for the "through-flow" reactor systems is for there to be a computer controlling at least one, and usually more than one, of the variable input parameters (such as the rate of input of a first start material; possibly the rate of input of a second start material; the bulk flow rate of the reagents in the reactor; the temperature of the reactor, (possibly at more than one zone), or the amount of reaction initiator/inhibitor), and a sensor providing information on an operational condition (for example, a pressure sensor sensing the pressure of the reagents or reaction in the reaction vessel; or a temperature sensor sensing the temperature of a region of the reaction vessel; or a viscosity measuring sensor monitoring the viscosity of the reagents or reaction product at some point of the reaction process).

The computer has a memory in which is stored, usually in a ROM memory chip, a pre-recorded correlation, for example a look-up table or chart giving the proper relationship between two operational parameters (for example, the pressure and the flow rate, or the temperature and the viscosity, or the temperature and the pressure). The sensor feeds

information about one of the operational parameters to the computer which checks it against the look-up chart and either knows what the other parameter of the chart is (for example because it controls it directly) or has the necessary information to allow it to know what it is. If the two values are either as they should be, or are within acceptable limits of where they should be if the reaction system is working properly, the computer takes no control action. If the two values effectively define a point off the pre-programmed parameter one versus parameter two graph that is held in the computer memory (or is not within the allowable error band) the computer takes action to vary one or more of the input parameters which it controls so as to modify the reaction operation such that the reaction conditions are brought back to the allowable expected ranges.

This feed back control operates in a matter of seconds. The sensors detect simple physical characteristics such as temperature, flow rate, or pressure.

Of course, there may be more than one sensor sensing the same, or different, operational conditions. The computer may have different look-up tables, or graphs, in its memory for different parameters.

The sensors are all "in-line", in the sense that they are making measurements on the reaction conditions experienced by the reaction products/ reaction reagents under the conditions of the reaction process: at the temperature of the process and using the process pressure to move the reaction materials around.

This form of "in-line" feedback control is successful.

A second control system for continuous flow reaction systems is where again a computer controls one or more of a number of input variable parameters (for example, the rate of input of a first and/or second input material; the bulk flow rate of material through the reaction vessel; the temperature of the reagents/reaction products, possibly at more than one zone), and signals indicative of the actual state of the product that is being produced are fed to the computer.

The second method differs from the first in that far more detailed information about the product produced is used to control the reaction process (rather than concentrating on the reaction conditions).

Viscosity measuring instruments, such as rheometers, are used to monitor the flow characteristics of a material as it is being extruded, or as it flows. Measurement of flow characteristics may occur as a continuous process by taking a bleed of flowing material and extrude it through a die of known geometry at a known flow rate, or pressure. Alternatively the process may be performed as a batch process in which samples of material are tested in isolation from processing of the material. Using the change in pressure over the die, the geometry of the die, and the flow rate through the die, the viscosity of the material can be calculated, usually automatically by the machine (shear viscosity is shear stress, derived from the pressure drop, divided by shear strain rate, derived from volume flow rate).

The viscosity of a material can alter with flow rate, and with temperature. In order to measure the changes in viscosity of a material over a large range,

or of different materials having different viscosities, it is necessary to use dies of different geometries - each die giving an accept‑ble "window" of measuring capability. To change a die in a rheometer it is necessary to remove an old die and replace it with a new die of different characteristics. Furthermore this involves a rheometer being used repeatedly to perform such measurements.

For a flow rate at any one temperature the rheometer can enable the computer to check that the viscosity of the polymer is correct (and hence know that the polymer being produced is in fact what was wanted). However, the viscosity of many polymers varies with flow rate, and with temperature. Therefore to be sure, by checking the reaction product, that what is being produced is what is wanted it is necessary to check the polymer at different flow rates and/or different temperatures. Since it is not practical to alter the entire production process by increasing the bulk flow rate (or alter its temperature) the rheometer takes a bleed from the main production line and has its own pump and temperature controller to make the bled polymer do what it wants, whilst the main bulk reaction proceeds under the unchanged parameters. Thus the rheometer pump can be set to drive the test polymer through a die at a variety of flow rates.

Gathering this more detailed information on the properties of the actual polymer produced takes a lot more time than the simple immediate parameter measurement of the earlier technique (for example about ten minutes). However, it does give more information about the actual product.

Once again the computer has a pre-programmed map of what it expects to see for the shear viscosity vs Flow Rate curve (or any other one parameter: second parameter curve), and if it sees that the measured curve is deviating significantly from the desired curve it controls the input parameters so as to cause the polymer produced to be more like the desired polymer. Clearly just what is controlled, and when, is pre-programmed into the computer.

Gathering the full-curve information takes something of the order of 10 minutes. This gives more information, and the user can be more sure that the reaction product is correct, but it introduces a delay in the control loop. It is only appropriate if it is safe to assume that the system stays stable over the 10 minute curve-generation time.

The more complex curve-gathering rheometer control method is not in-line in the sense that the motive power pushing the polymer being tested comes from the reaction process: it is on-line in that it is attached to the on-line continuous reactor, but has its own motive force generating means so that polymer can be made to experience conditions that are not present in the continuous reactor.

In one known kind of analysis of a processed solid material using a simplified rheometer of the kind known as a melt flow indexer, the material is provided in granule or powder form and is pushed into a melting cylinder which is usually a steel barrel. The material is usually tamped down into the heated barrel which melts it. Heating elements are provided associated with the barrel. A melt typically occupies approximately 60% of the volume of the granulated or

powder material. A piston pushes the melt through a capillary die provided at the bottom of the barrel.

The material is tamped down to increase thermal conduction between granules of material to improve the rate of melting, and to improve the uniformity of the melt.

After each measurement operation traces of material may be left in the barrel. The traces may be present in a consistency or a form falling within a range extending from an oil-like material to a toffee-like material. Residue of material left in the barrel may cause blockage of the rheometer the next time it is used or contamination of the next sample which may, of course, be a material of a different composition. Therefore the barrel must be cleaned after each measurement operation. This may, for example, require a cleaning operation every ten minutes whilst the rheometer operates. The rheometer may be in operation all day or longer. The cleaning operation is conventionally performed manually.

In conventional melt flow indexer rheometers the operation of a piston pushing melt material through the die, removal of the piston from the barrel, cleaning of the piston barrel and die, and insertion of another batch of material into the barrel and tamping of the material are performed manually, sequentially, and in that order. This is time consuming.

The heating elements in rheometers are conventionally disposed around the barrel. It is critical to the determination of flow characteristics that the temperature of the melt be uniform and accurately determined. Heat can escape by radiation and

convection from the lower end of the barrel. Although heating elements extending to the bottom of the barrel may alleviate the problem of heat loss, this may also interfere with insertion and removal of the die. One known expedient is to insulate this area with PTFE insulators. Whilst this is reasonably effective it does limit the upper working temperature of the rheometer to 250°C. Above this temperature PTFE degrades thermally.

According to a first aspect of the invention we provide a rheometer comprising a die, a material holding chamber, and cleaning means; in which the cleaning means is adapted to clean the material holding chamber automatically.

Preferably the cleaning means comprises a plug and means for urging the plug through the material holding chamber so as to clean the chamber. The plug may be cylindrical. The plug may have a generally uniform outer surface. The plug may have a rough or smooth surface. The plug may have a stepped surface having raised and sunken regions adjacent one another. Preferably the plug has at least one raised region adjacent a sunken region. There may be a sunken region to either side of the raised region. A raised region may be a ring. A sunken region may be a ring. The plug may have the form of a series of rings of alternating wider and narrower diameter.

The cleaning plug may be provided in delivery means adapted to deliver the plug to the entrance to the rheometer bore. The delivery means may comprise a plug-holding bore in a body, the plug being provided in the plug-holding bore before it is introduced into the

rheometer bore. The plug-holding bore may be a through bore.

Preferably the material holding chamber is an elongate bore, most preferably defined in a barrel.

The cleaning means may be adapted to change diameter when inserted into the material holding chamber. The plug may be resilient. It may be adapted to be compressed from a wider diameter to a narrower diameter. Alternatively the plug may be adapted to expand in the material holding chamber. This may be achieved by thermal expansion of the plug, or a part of the plug, when exposed to an increase in temperature. The plug may be of complementary shape to the material holding chamber so as to be a tight interference sliding fit in it. The plug may be adapted to thermally expand in the material holding chamber such that the outer surface of the plug is in bearing contact with the inner surface of the material holding chamber. The plug may comprise a body portion and a split ring surrounding a region of the body portion. The plug, or elements of the plug adapted to be in contact with the material holding space, may be made of phosphor bronze.

Alternatively the plug may be made of wood, or plastics or polymeric material such as "Tufnol" (Trade Mark) polymer.

The plug may comprise an elongate body having one or more split rings, such as piston rings, mounted on it.

The plug may be re-usable or disposable after use.

The plug may be a spring. The spring may be helical. The coils of the spring may be adapted to bear against the inner surface of the material holding chamber when located therein. The length of the spring may be reduced, in order to achieve radial expansion, when it is in the chamber to be cleaned. Some other manipulation of the spring may be used to expand it radially (for example it could be twisted).

The plug may be provided additionally with flexible cleaning material. The flexible cleaning material may be cotton, for example a pad or a patch, or a covering.

We envisage pushing or pulling the cleaning plug through the material holding chamber.

The plug may comprise an invention in its own right, and could be used with automatic cleaning or manual cleaning.

According to a second aspect of the invention we provide a method of operating a rheometer comprising the steps of pushing melt from a material holding space through a die with motive means, removing the motive means from the material holding space, cleaning the motive means, and refilling the material holding chamber; in which the operations of refilling of the holding space and cleaning of the motive means overlap in the same period in time.

Preferably the cleaning operation and refilling operation are automatic.

According to a third aspect of the invention we provide a rheometer comprising a material holding

space, die means, tamping means, and motive means adapted to push material from the material holding space through the die; in which the tamping means and the motive means are different components.

Having the motive means as a separate component from the tamping means enables us to clean the motive means whilst the tamping means is performing its function.

The motive means and the tamping means may both be elongate. They may extend in the same direction. They may comprise elongate rods.

Preferably the tamping means and the motive means may be moved simultaneously. Preferably the tamping means and the motive means are both mounted on the same mounting member. Preferably the mounting member is swivelable. Preferably the mounting member is adapted to index from a first position to a second position. In a first position the motive means may be located above the material holding space. In a second position the tamper may be located above the material holding space. Preferably in the second position the motive means may be at a cleaning station to be cleaned.

Alternatively, indexing of the motive means and the tamping means to and from respective operative positions and inoperative positions may be achieved with the tamping means and the motive means mounted on different components.

Preferably the motive means is a piston, and most preferably a piston and load cell in combination.

The cleaning and tamping may be done automatically or manually.

According to a fourth aspect of the invention we provide a rheometer comprising a die means and a material holding space; in which heating means is provided adjacent the die.

Preferably the heating means is provided below the die. Most preferably the die has a lower face and the heating means is provided beneath the lower face of the die.

Preferably the material holding space is a rheometer barrel.

The heating means may comprise one or more heaters. Preferably one or more cartridge heaters are provided below the die.

The heating means may compensate for heat loss from the bottom of the rheometer barrel.

The heating means below the die is preferably controlled by a controller which receives temperature signals. The temperature signals may include signals from the region of the die or the bottom of the material holding space.

According to a fifth aspect of the invention we provide a method of filling a material holding space in a rheometer comprising the steps of loading the material holding space with a quantity of material, tamping the material, and loading the material holding space with a further quantity of material.

Preferably several tamping operations are performed. Most preferably a tamping operation is performed after each loading of the material holding space with a charge of test material (less than the volume of the holding space). A series of tamping operations may be interleaved with a series of loading operations.

Preferably loading means may be used to load the material holding space. The loading means may comprise a carousel. The loading means may comprise a plurality of blocks. A block may comprise a plurality of supply cells. There may be about three supply cells in one block. There may be a plug-holding cell provided in the carousel, preferably in the block. The material holding space may require the contents of several cells before a test operation can be performed. The holding space may be capable of receiving the contents of an entire block of cells.

Several tampings provide a greater degree of uniformity to the compressed powdered or granulated material. Non-uniformity of material in the material holding space may be a considerable source of error.

According to a sixth aspect of the invention we provide a rheometer comprising a die, a material holding space, tamping means, sensor means adapted to measure a parameter of the rheometer, and control means adapted to control the operation of the tamping means in response to the measurement of the parameter.

The parameter of the rheometers may be pressure or load xerted by the tamping means. The sensor may, however, measure conditions in the material or even pressure or force exerted on the material holding space.

Preferably the control means is adapted to respond to a specified value, for example load. The control means may change the operation of the tamping means. The change in the operation of the tamping means may be a change from tamping to not tamping, or it may be to reduce the pressure or load exerted by the tamping means.

It is intended that by controlling the tamping force greater uniformity of material may be achieved. We may prefer to ensure that the test material is tamped to a predetermined load or pressure. We may ensure that the tamping load or pressure does not exceed the predetermined level.

Preferably heating occurs during the tamping operation.

The tamping pressure may be applied for a controlled predetermined time. The control means may control the tamping means to apply one of a number of predetermined tamping pressures for one of a known number of times.

According to a seventh aspect of the invention we provide, in a rheometer, die transfer means which is adapted to collect a die from a rheometer, and transfer the die to a cleaning station.

Preferably collection means are provided to collect a die from a rheometer. The collection means may be a scoop or cup. The collection means may also be adapted to collect cleaning means, such as a plug, according to a first aspect of this invention. Preferably the collection means is mounted on indexing means, such as an arm, which is adapted to index the

collection means from the rheometer to the cleaning station.

The cleaning station may be a furnace. The cleaning may be thermal cleaning. Thermal cleaning may occur by burning away a quantity of material on a die. The furnace may have an extraction system to remove exhaust products from combustion of material.

According to an eighth aspect we provide a rheometer having automatic die changing means.

One advantage of this is that it enables the die through which the material being tested is flowing to be changed whilst the rheometer is still on-line - whilst the extruding or flow-generating of a main material dispenser is still working. Furthermore, since the die can be changed automatically we can replace a die which is, for example, blocked or partially blocked, with a fresh die in a matter of seconds, rather than minutes as was required to change a die manually and without the need for personnel to be present. However, the invention can be used with the rheometeras described in relation to the drawings, or a simplified melt flow indexer which derives from it.

The die may be changed for a similar or identical die, or it may be changed for a die having different characteristics. Changing the die for one with different characteristics enables us to test the material for different properties, or for a greater range of the same property, possibly (when used on-line) in the same operational run of the main xtruding or like flow-generating apparatus from which the flow to the rheometer is taken.

Preferably the die changing means is controlled by automatic control means, such as a microprocessor, computer or the like.

The automatic die changing means preferably has a plurality of interchangeable dies of different characteristics. The rheometer can change one die from being in an operative condition to an inoperative condition, and the previously inoperative die to being operative, thereby varying the flow characteristics of the die being used.

Preferably the automatic die changing means comprises a cartridge or block having a plurality of dies and a corresponding plurality of operative positions with respect to a flow passageway of the rheometer with which the operative die is in communication. Cartridge moving means are preferably providing to move the cartridge between its operative positions. The cartridge moving means is preferably under the control of the automatic control means. The cartridge of dies preferably has dies arranged in a line, or around an arc of a circle. Cleaning plug holding means may also be provided in the cartridge.

The rheometer preferably has seal means to seal the die that is in use to the flow passageway. The seal means preferably includes clamping means adapted to urge the die against an abutment face provided on the rheometer. The clamping is preferably under the control of the automatic control means.

The rheometer may have sensing means to detect automatically when a die is blocked, the control means then preferably changing the die automatically.

According to a ninth aspect of the invention we provide a rheometer comprising a die, a material holding space and cooling means; in which the cooling means is adapted to cool the material which emerges from the die.

Preferably the material is cooled to solidify the material.

The cooling means may be one or more air jets or fans.

The cooling means may be an air ring.

Preferably the diameter of the material is measured after the material has been cooled by the cooling means. This effectively "freezes" the swell of the material as it emerges from the die and enables die swell measurements to be made more easily. The cooling means is preferably close to the die, and most preferably immediately below the die heater.

According to a tenth aspect of the invention we provide a rheometer comprising a die, a material holding space, and cutting means; in which the cutting means is adapted to cut the material which emerges from the die when the extrudate reaches a pre-determined length.

Means to vary the predetermined length may be provided.

Sensor means may be provided to measure the length of xtruded material. The sensor means may be adapted to operate the cutting means automatically. Operation

of the cutting means may occur in response to a signal from the sensor means.

In having pre-determined lengths of extruded material, measurements carried out on the material may be more accurate. The measurements may include the diameter of the extruded material at any point along its length. Accordingly, diameter measuring means may be provided. This may enable the swell of the material to be determined.

The cutting means may cut material to provide length of about 70mm. The cutting means may again operate once the total length of material is a second known length, for example about 120mm. Diameter may be measured with diameter measuring means which may then be movable out of the way. The diameter measuring means may operate only when the extrudate has a predetermined length, or one of a number of predetermined lengths.

Instead of measuring the diameter of the extrudate some other parameter indicative of swell might be measured. For example, the weight of a predetermined length of extrudate might be measured.

The ninth and tenth aspects of the invention may be combined together in a machine to cool, and perhaps solidify material, which may then be cut to provide pre-determined lengths of material.

Measurement of the swell of the material provides information concerning the elastic properties of the material. Once emerged from the die the material may have a viscosity within the range from an oil to a solid. It is important to define a certain length of

extruded material in order to determine the weight of the extruded material, and hence the load at the point of diameter measurement.

According to an eleventh aspect of the invention we provide a rheometer comprising a die, a material holding space, and cutting means; in which the cutting means is adapted to hold the extruded material against lateral movement during cutting of the extruded material.

Preferably the cutting means comprises pincer cutters. The cutting means may have a jaw region which is adapted both to hold and to cut.

According to a twelth aspect of the invention we provide chemical reactor apparatus comprising test means adapted in use to test a reaction product, or for testing an intermediate reaction product, reaction condition means adapted in use to detect one or more reaction condition, and control means adapted in use to control at least one reaction input parameter; in which in use the reaction condition means provides to the control means a signal indicative of at least one reaction condition, and the test means provides the control means with a second signal indicative of a property of the reaction product being produced, and in which the control means utilises both the first and the second signals in controlling the reaction input parameter.

We are the first to appreciate that the known two control systems can be combined. In one embodiment we use a long term control loop to modify the "target" curve for a short term control loop.

Preferably the reaction condition means produces a first signal indicative of the pressure at a region of the reactor apparatus; or an indicative of the temperature at a region of the reactor vessel; or of the bulk flow rate of material in the reactor vessel; or of the speed of drive of drive means for the reactor vessel; or of the amount of, or ratio between, first and second start materials being input to the reactor vessel.

There may be reaction condition means provided to produce a signal indicative of any one of the above, or any pair of the above, or any combination of the above. The reaction condition means may be a single sensor (such as a combined pressure and temperature sensor), or it may comprise a plurality of sensors which detect different physical conditions. There may be more than one sensor to detect the same physical condition; they may be at different locations.

The test means may provide signals indicative of the shear viscosity of the material being tested; or of the die swell of the material; or of the extensional viscosity of the material; or of the wallslip of the material. The test means may provide signals indicative of any pair of the above characteristics of the material, or any combination of the characteristics. More than one sensor may be provided.

The test means preferably comprises a die. The die preferably has a plurality of holes of different sizes (eg different lengths, or different diameters; or a set of holes with different diameters and a set with different lengths).

Preferably the control means responds to signals from the reaction condition means as a short term control of the reaction conditions.

Preferably the control means responds to signals from the test means as a longer term check on the characteristics of what is being produced. Preferably signals from the test means are used by the control means to amend or alter a reaction condition look-up table or parameter one: parameter two graph held in a memory of the control means, said reaction condition graph being used by the control means to control said reaction input parameter in response to signals from said reaction condition means.

Thus in one embodiment of the invention we can have a parameter one : parameter two look-up table/graph in RAM, and alter the table/graph in response to signals indicative of the properties of material being tested (as opposed to conditions of the reactor vessel, which might not necessarily amount to the same thing).

We could, therefore, initialise a polymer production reactor (for example) by running it at approximately the right conditions required to produce the desired polymer, and then adjust input parameters (such as rate of feed of feedstock chemicals, ratio of inhibitor to initiator, temperature, until our test means was showing that the polymer produced has the desired characteristics and then automatically enter the short-term physical condition parameters required to achieve this, together with a parameters one: parameter two table/graph for each condition parameter that is part of the control process (these may be a first approximation from a standard source, eg program

or chip). The curves are most likely to have a known general form. The reactor would then be controlled in the short term by signals from the reaction condition means being compared with the short term physical condition parameter graphs and the control means taking action to get the conditions closer to the set curve (if necessary). The material test means would periodically check the characteristics of the material produced and would compare the measured characteristic curve with the initialised curve, and if adjustment of the measured curve is required the control means would amend the short term physical condition parameter graphs so as to achieve the desired result.

Of course, instead of initialising the long term material physical characteristic curve by a test run, this could be input manually (for example by keying in the initial curve, or by plugging in an appropriate memory, or by transferring electronic information to the control means.

One advantage of the invention is that if the starting materials change (for example, different chemical formulae feedstock is used) there is not need to re-enter all of the immediately measured parameter one: parameter two curves: this may in one embodiment be done by the apparatus itself as it self-corrects.

Furthermore, if the characteristics of the polymer being produced are to be changed, there is no need to scrap the existing look-up tables and re-enter them manually. The long term material characteristic curve is adjusted (either from an external source or by trial run) and the reactor automatically re-initialises so far as the reaction condition parameters are concerned. This can be a considerable saving.

A further advantage is that if, for whatever reason, the physical characteristics of the material being produced change or drift over time, despite the input reaction conditions being maintained (for example if there is a gradual build up of unreacted initiator) then the test means can compensate for that automatically.

Preferably the control means has a memory to which is written, at least in one mode of operation, signals from the test means, or the reactor condition means, or both.

The reaction product may be produced and checked by the test means, the input parameters required to achieve a deserved result being written to the memory.

The control means may check or update its reaction condition parameters periodically, for example at about 10 minute intervals. The control means may monitor or control its input reaction parameter at about 1 second, 2 second, or 3 second intervals.

Preferably the control means monitors and/or corrects its input reaction parameter at intervals of a first time period, and updates or monitors the look-up table/graph that it uses to monitor its input reaction parameter at a second, longer, interval. Preferably the second time interval is at least an order of magnitude longer than the first time interval, and it may be two orders of magnitude longer, or even three or four orders of magnitude, or more, longer. The second time interval may be about 600 times longer than the first time interval.

According to a thirteenth aspect of the invention we provide a method of controlling a production reactor adapted to produce a production material from one or more starting materials, the method comprising the steps of having a look-up table, graph or other concordance between a first physical condition measured in the reactor or input to the reactor and a second physical condition measured in the reactor or input to the reactor; using the look-up table or other concordance to control an input parameter of the reactor so as to try to maintain stable the nature of the production material; testing one or more physical characteristics of the production material; and using the results of the testing of the physical characteristics of the material to modify the look-up table or other concordance, or to modify how that table or concordance is used in the control of the input parameter.

The invention also comprises, in a fourteenth aspect, a control system for reaction apparatus, the control system being adapted to enable the reaction apparatus to be in accordance with the first aspect of the invention, or to operate in accordance with the second aspect of the invention.

Looked at in one way we provide a reactor having a control response table/curve for use in controlling physical conditions of the reactor which is in RAM and which is capable of being updated automatically by test means which test the actual material being produced.

According to a fifteenth aspect of the invention we provide a control system for a reactor having test means for testing characteristics f the material being produced by the reactor, and also having condition

sensing means for detecting conditions being experienced in the reactor; signals from the test means and from the condition sensing means both being used in the control of the reactor.

According to a sixteenth aspect of the invention we provide a reactor system having an in-line sensor and an on-line sensor.

In the preceding paragraph an "in-line" sensor is one in which material is moved past the sensor by the reactor's drive means, as opposed to any independently controlled drive means; and an on-line sensor is a sensor in which material is moved past the sensor by separate drive means that are controllable independently of the drive means of the reactor that produce bulk flow through the reactor.

An embodiment of the invention will now be described by way of example only, with reference to the accompanying drawings of which:-

<u>Figure 1</u> shows a side elevation of a rheometer system according to the invention;

<u>Figure 2</u> shows in greater detail part of the rheometer shown in Figure 1;

<u>Figure 3</u> shows schematically a plug cleaner for a rheometer;

<u>Figure 4</u> shows schematically an embodiment of a plug;

<u>Figure 5</u> shows an alternative embodiment of a plug;

<u>Figure 6A</u> shows a rheometer having a die heating system;

<u>Figure 6B</u> shows schemetically the relationship between a die and a slotted plate which holds it in place in the barrel of the rheometer;

<u>Figure 7</u> shows a rheometer having a simultaneous tamping and piston cleaning system;

<u>Figure 8</u> shows a material loading arrangement having a plurality of loading blocks;

<u>Figure 9</u> shows a schematic load and temperature control system for a rheometer;

<u>Figure 10</u> shows a rheometer having an extruded material cooling system;

<u>Figure 11</u> shows a pincer cutter, typically for use with the system of Figure 10;

<u>Figure 12</u> shows schematically the die changing means of the rheometer of Figure 1; and

<u>Figure 13</u> shows a side view of the die changing means of Figure 12.

<u>Figure 14</u> schematically shows one prior art control system for a reactor for producing plastics polymer materials;

<u>Figure 15</u> shows a Pressure vs Flow Rate control graph stored in ROM in the control processor of the system of Figure 14;

Figure 16 shows a second prior art control system for a reactor for producing plastics polymer materials;

Figures 17a to 17d show schematically graphs of property one vs parameter two produced by the system of Figure 16, and used in the control of the system;

Figure 18 shows schematically a new reactor control system for a reactor adapted to produce plastics polymer material;

Figure 19 shows a graph of property one vs parameter two relating to the system of Figure 18;

Figure 20 shows schematically the modification in the system of Figure 18 of a graph of condition parameter one vs condition parameter two used in the control of the system of Figure 18; and

Figure 21 shows schematically a die used in a rheometer of Figure 18.

Figure 1 shows a rheometer system 2. The rheometer system 2 has a frame 4, a material supply system 8, a force applying system 10 with drive means 12, a die changing means 14, a gas purge system 15, and a furnace 16.

The rheometer is shown in greater detail in Figure 2. It comprises a steel barrel 18 having a bore 20 running along the length of the barrel 18. The barrel 18 has a countersunk lead-in 19 to guide into the bore material to be tested or a piston. Surrounding the barrel 18 is a heater 22. The heater may be a

single heating unit or a composite of several heating units as shown in this embodiment.

As seen in Figure 2, a die 24 is held at the bottom end of the barrel 18 so that the bore 20 is in communication with an aperture in the die. The die 24 is held in place by a plate 25. The plate 25 has a key-hole slot with a wider portion and a narrower portion. The plate can be slid sideways by a solenoid (not shown) and when the wider portion of the key-hole slot registers with the die the die drops out of the barrel. This can best be seen in Figures 6A and 6B.

The rheometer is provided in a chamber 7 to which the gas purge system 15 is connected. The gas purge system is adapted to pump nitrogen, or some other inert gas, into the chamber. This is used when testing some materials which react in air and enables us to test materials in an atmosphere in which they do not react or degrade.

The material supply system 8 is shown in Figures 1, 2 and 8. Referring mainly to Figure 2, the material supply system 8 is located above the rheometer barrel. A carousel disc or cylinder 26 has a series of slots 28 extending inwardly from the peripheral edge of the circular carousel 26. Supply blocks 30 are located in respective slots and project above them. Each of the blocks 30 may be moved radially with respect to the carousel 26. The blocks 30 each comprise a series of individual supply cells 34, 36, 38 adjacent one another in a row forming the block 30. The cells are aligned radially along the carousel 26 with cell 34 farthest from the centre of the carousel 26 and cell 38 n arest to the centre. In addition to the supply

cells 34 to 38 there is a cleaning plug cell 32 which contains a cleaning plug, referenced 84.

Each individual turn of the carousel 26 indexes a block 30 to register with the countersunk lead-in 19 of the barrel 18. The block 30 may move radially with respect to the carousel in a series of steps. Each step registers an individual load cell with the countersunk lead-in 19.

The supply cells 34, 36 and 38 have an open upper end but their lower ends are blocked by a plate portion of the carousel 26 (referred to in Figure 2 as numeral 25). The lower plate portion 25 of the carousel 26 has a series of through-holes 27, one in each slot 28, which register in turn with the lead-in 19 to the bore 20 as the carousel is indexed around. In this embodiment, once a supply cell is in registration with the rheometer bore the material is free to fall from the cell to the bore. The material may be pushed out by the tamping piston. A similar arrangement is provided for the cleaning plug cell 32.

In an alternative embodiment a slidable plate or grate may be located at the bottom of the supply block 30 to close the bottom end of all of the supply cells. Once a supply cell is in registration with the rheometer bore, the plate is moved to open the end of the supply cell and the material is free to fall from the cell to the bore. Of course, each supply cell may be provided with individually moveable plates or grate which may be operated independently.

The material supply system 8 is disposed adjacent to the rheometer barrel on one side. Disposed to the

other side of the rheometer barrel is a piston and tamper cleaner 80.

The die changing means 14 is located below the level of the bottom end of the rheometer barrel, as is shown in Figures 1 and 2. The die changing means 14 is also shown in greater detail in Figures 12 and 13. The die changing means 14 comprises a transport arm 40, a loading ring 42, a loading cylinder 44 and an ejection cylinder 46.

The transport arm 40 is driven to rotate through 360° about an axis 48 passing through an end 150 of the arm 40. In rotating through 360° the arm pass underneath the rheometer 6, through the furnace 16 and underneath the loading ring 42.

Figure 13 shows the relative positions of the transport arm 40, the ring 42 and the cylinders 44 and 46. The ring 42 overlaps the transport arm 40. The cylinder 44 has a loading piston 52. The cylinder 46 has an ejection piston 54. The cylinders 44 and 46 may be electrical servos, or pneumatically or hydraulically driven (or driven in any other suitable way).

Referring back to Figure 1, the drive means 12 comprises means for producing movement of the rheometer test, or loading, piston 70 and the tamping piston 72. In this embodiment there is a screw jack and drive motor 56. The force applying system 10 is adapted to apply force to a cross head 58. The cross head runs on guide and support bars 60, 62. Mounted on the lower face of the cross head 58 is a plate 64 carrying load cells 66 and 68 which measure load in the rheometer test piston 70 and the tamping piston 72 respectively. The pistons 70 and 72 are guided and

supported by a support strut 74 which is guided by a guide sleeve for movement along a support bar 76 (which in turn is carried by the head 58).

The plate 64, load cells 66 and 68, pistons 70 and 72 and strut 74 together form a swivel unit 78 which is capable of swivelling about the support bar 76. This unit is shown in Figure 7. The drive means for the swivel unit 78 is not shown in the drawings.

The swivel unit may swivel between a first position in which the loading piston is registered above the rheometer barrel and a second position in which the tamping piston is registered above the rheometer barrel.

Located beneath the rheometer barrel is a diameter gauge 122 which is moveable from a first position (referenced A in Figure 2) to a second position (referenced B in Figure 2). This is shown in Figure 1 and in slightly more detail in Figure 2.

Figure 3 shows schematically an embodiment of a barrel cleaner according to one aspect of the invention. A plug 84 is held over a bore 83 of a barrel 86 of a rheometer to clean the barrel 86. The plug is provided in a cartridge 87 which also contains sample to be tamped down (not shown). A piston 85, which in this embodiment is the tamping piston (but may be some other piston) pushes the plug through the bore 83 to clean it. The plug drops out of the open bottom end of the bore.

As a further example, in an alternative embodiment of the invention the swivel unit shown in Figure 7 may have a third pist n mounted on the plate 64. In this

alternative embodiment the swivel unit 78 may be adapted to swivel to an additional third position in which the third piston is adapted to be swivelled into a position above the rheometer 6.

Figure 4 shows an embodiment of the plug 88. A plug body 90 comprises an end stop 92 and a series of annular discs 94 having alternating wider and narrower diameters. The end stop 92 and the narrow diameter discs 96 are comprised of stainless steel. The wide diameter discs 98 are comprised of phosphor bronze. A screw threaded rod 100 extends from one face of the end stop 92. The discs are threaded onto the screw threaded rod 100 in the order described above. The discs 94 may simply be washers able to rotate on the screw thread or may be internally screw threaded to cooperate with the screw threaded rod 100 and to lock firmly into place against one another. A retaining disc 102 is screw threaded to hold the other discs in place.

In a production version the plug may be a single piece of bronze, it may have the same geometry as that shown in Figure 4, but be turned from an integral piece of bronze.

As seen in Figure 2, the plug 84 cleans the bore 20 by scraping or wiping off any smudging of residue which may remain following a test operation. The plug has such a tight fit with the bore that any test material that may be left on the sides of the bore is swept away by the plug.

Of course, it will be appreciated that the test piston should sweep the barrel clean as far as possible, the die should be removed, and then the

tamping piston will push the cleaning plug through to clean the barrel.

After a measurement operation using the rheometer 84 it will usually be desired to remove the die at the bottom of the rheometer barrel 83. Residue of the test material extruded may remain along the bore 83 of the barrel 86. A cleaning plug as described in relation to Figure 4 is brought into a position above the barrel. The plug is then inserted into the barrel 86. A countersunk lead-in at the entrance to the bore 83 may guide the plug into place.

The barrel 86 will still be hot and this heat will increase the temperature of the plug 88. Phosphor bronze is a material having a relatively high thermal co-efficient of expansion. Since the operating temperature range of the rheometer will be known as well as the diameter of the bore 83, a suitable diameter of phosphor bronze disc may be determined to ensure a close fit between the discs 98 and the bore 83 once the plug has fully warmed up. Even if the discs 98 are an interference fit in the bore 83, phosphor bronze is a good bearing material, and the disc should be moveable longitudinally with respect to the bore 83. The plug is pushed downwardly through the bore scraping off residual sample material as it progresses. The plug 88 may be pushed completely through the bore 83 to carry as much sample material as possible. The plug may not be attached to the rod 85 (or may be releasably attached) and preferably falls off (or is removed from) the rod 85 once it emerges from the bottom of the barrel 86 (the die having first been moved out of the way). The plug 82 is collected at the lower end of the rheometer barrel 86 as it emerges.

It may be desirable to choose the diameter of the discs 98 such that on thermal expansion in the bore, the first to enter is a close fit, the second to enter is a near interference fit, and the third to enter is an interference fit, in order to provide progressively better cleaning of the bore. Of course the plug may carry more or less than three discs.

Figure 5 shows an alternative embodiment of a cleaning plug. The plug comprises a helical spring 104. The spring is of a width slightly greater than a bore 83 and when inserted in a bore, the coils of the spring 104 press outwardly against the bore 83. Since this embodiment would provide a rather inexpensive plug, such a helical spring plug may be disposable.

In each embodiment the principle is the same: that when the plug is inserted into the bore 83, surfaces of the plug apply pressure to or lie in close proximity to the internal surface of the bore 83. Other plugs may be made of wood, ceramics, or any other suitable material.

Another feature of the rheometer system 2 not previously described is shown in Figures 6A and 6B. Since Figures 6A and 6B are schematic the feature will be described in relation to a third rheometer, but it will be appreciated that it is also present on the embodiment of Figure 1. The rheometer 89 of Figure 6A has a barrel 86 provided with a bore 83. A heater 106 is shown surrounding the barrel 86 and a die 108 is shown located at the bottom end of the barrel 86. A sliding plate 91 holds the die in the bore. The plate 91 has a key-hole slot (or some other arrangement with a hole wide enough for the die to pass through). A further heating system 110 is located adjacent to and

beneath the die 108. The heating system may comprise one or more cartridge heaters 111. Heating in this area beneath the bottom face of the die compensates for heat loss through the base of the barrel. This is intended to give a more uniform temperature throughout the barrel 86 to provide accuracy of temperature readings. Moreover, since the test material previously flows through the die in the region of the die we control the temperature of the very region where the flow characteristics are important.

Figure 10 shows a further embodiment of the invention. The arrangement of a barrel 86, a heater 106, and a die 108 is maintained. Additionally a cooling system 112 is included below the die to cool extruded material which emerges from the die. If the additional heating system as described above is included in this embodiment, it may be provided between the bottom of the die 108 and the cooling system 112.

The cooling system uses jets of air to cool extruded material so that it sets. The cooling system is preferably an air ring which surrounds an extruded string of test material.

Again the embodiment of Figure 1 has this feature, but it is not described or shown in Figure 1.

Figure 9 shows schematically a control system for providing more uniform tamping. A tamping piston 114 is driven by drive means 116. A load cell 118 measures the load exerted by the drive means 116 on the tamping piston 114. A controller 120 controls the force applied by the drive means and receives a signal from the load cell 118 indicative of the force. Tamping is generally

performed as a multi-stage operation. As discussed earlier supply cells 34, 36 and 38 sequentially provide a quantity of material to the rheometer bore. After a quantity of material is supplied it is tamped down with the tamping piston 114. Once a pre-determined force is detected by the controller 120, the drive means 116 stops applying force (possibly after maintaining that force for a period) and the tamping piston 114 is removed. The rheometer is heated and the material is melting. A further quantity of material is supplied by another supply cell and tamping force is applied until another pre-determined force is detected and again the tamping piston 114 is removed. This procedure continues until the rheometer barrel is fully loaded. By carefully controlling the force applied during tamping the distribution of material and the properties of the material, and distribution of packaging density through the rheometer, is controlled and is more uniform. Uniformity of these parameters allows for greater accuracy in measurement of rheology properties of the material.

Again the embodiment of Figure 1 has this feature, but it is not shown or described.

A discussion of the operation of the rheometer system 2 of Figure 1 follows. Reference may be made to Figure 2 which shows some details more clearly. At the point where we join the test cycle the rheometer barrel has been cleaned and a clean die is provided at the bottom of the empty rheometer barrel. The piston 70 which has just forced test material out of the rheometer is not yet clean and still has material from the last measurement operation adhering to it.

The carousel 26 indexes around to bring one of the slots 28 into alignment with the top end of the bore 20 of the rheometer barrel 18. A supply block 30 is moved along the radius of the plate to bring the first of the supply cells into registration with the rheometer bore 20.

Material is loaded into the barrel sequentially from each supply cell by the tamping piston 70 moving downwards (carried by the head 58) and pushing it out. The tamping piston 70 is, of course, first swivelled to register with the bore 20.

After each quantity of material is supplied to the rheometer bore the supply block 30 is moved away from the rheometer barrel and the tamping piston 70 is brought down to tamp the material in the barrel. The control system shown in Figure 9 operates to provide uniform tamping in the rheometer barrel.

The force and motion to the tamping piston 70 is also applied to the loading piston 72 since it is the head 58 that is moved. As each tamping operation occurs and the tamping piston is inserted into the rheometer barrel, the loading piston 72, in an uncleaned state is also brought down. The loading piston is brought into the cleaner 80 to one side of the rheometer barrel. The cleaner operates to clean the loading piston 72. Accordingly the test piston is cleaned whilst tamping occurs.

The cleaner 80 comprises a mechanism for cleaning the tips of the test piston and/or tamper piston. For example, one or more rotating brushes. The cleaner 80 cleans the bottom end face of the test piston 72.

Once the material is in rheometer barrel is fully loaded and tamped in the rheometer barrel, and has melted, it is ready to be tested. The swivel unit 78 swivels about the bar 76 and the test, or load, piston 72 is located above the rheometer barrel and the tamping piston 70 is located above the cleaner 80. The drive means 12 then pushes the test load piston 72 into the rheometer barrel and material is extruded out of the die.

As mentioned above, the die is usually heated by heating system 110 to ensure accuracy and uniformity of the temperature of the material. Thêre is, of course, a controller and sensors for the heating system, with appropriate feedback.

The diameter gauge 122 is located in a position beneath the die. As a length of extruded material extrudes out of the die the diameter of the length of extruded material is measured periodically by the diameter gauge. The information is fed to a central processor which determines the die swell from the diameter of the aperture in the die, the length of the extruded material and the diameter of the length of extruded material. As the length of extruded material grows it passes through the cooling system 112 where it is cooled and solidified.

When the extruded material reaches a length of 70mm a pair of pincers close to cut off the end of the length of material. The action of the pincers may be initiated in response to a signal provided by a sensor which detects the extruded string of material. The length of material is now a standard length and any measurement of the diameter of the extruded material close to the die has a repeatable, consistent, effect

due to the effect of gravity on the length of the material (effectively we can calculate the load on the hanging string of extrudate at the point of measurement, measure its diameter, and calculate swell).

If we vary the length of the suspended string (by varying the position of the sensor or length cutter, or both) we can see how the swell depends upon the load. This can provide useful information.

Figure 11 shows a pair of pincers 124 for cutting the string of extruded material to a predetermined length.

The pincers 124 are comprised of two components 126 and 128 which are pivotally attached at a pivot point 130. At one end of the pincers 124, cutting surfaces 125 and 127 on the components 126 and 128 surround a cutting region 132. The cutting action of the pincers is operated at the other end of the pincers 124. As the ends of the components 126 and 128 furthest from the cutting region 132 are brought together as shown by the arrows 134 in the drawing, the cutting surfaces are brought together. A length of material present in the cutting region 132 will be pinched by the cutting surfaces 125 and 127 and held in place against lateral movement whilst the end of the length of material is cut off. This avoids us forcing the string of extruded material sideways, as we would do if we used straight-bladed scissors.

The length of material continues to grow after the end has been cut off, and measurements of the properties of the material continue. Once the length of material is 120mm long, a sensor detects the end and

the pincers are operated again to standardise the length of material to 70mm. Thus, we get diameter measurements at two known loads - 70mm and 120mm.

The system has a sensor 103 to detect when the string reaches 120mm (length D of Figure 2).

Both the length C and the length D can be varied under the control of the user, either automatically adjustable or manually adjustable. This enables us to have strings of extrudate of different known lengths and enables useful data to be obtained.

Returning to the operation of the general overall machine, as the loading piston moves deeper into the rheometer barrel, the tamping piston enters the cleaner 80 where it is cleaned of material remaining on its surface. Accordingly the tamper is cleaned whilst the loading piston is acting on material in the rheometer barrel.

At the end of the extrusion and measurement operation, the test load piston is withdrawn from the rheometer barrel. The diameter gauge 122 and cooling ring 112 are moved to a position to the left of the bottom of the rheometer barrel 18 as shown in Figure 2. The plate 25 is slid sideways by its solenoid to align the wider part of the key-hole with the die and the die falls off to be received in hole 137 of arm 40. The transport arm 40, carrying the used die, indexes around, removing the used die from the vicinity of the rheometer barrel. A plug collecting portion (hole) 136 of the arm 40 is brought into register with the bottom of the rheometer barrel. The carousel then brings a new block 30 into line with the bore of the barrel and a cleaning plug 84 is then driven through the rheometer

barrel by the tamping piston 70 to clean it as set out above. The tamping piston 72 is used to drive the plug. The plug passes straight through the barrel and falls into the plug collecting portion 136 of the arm 40.

Referring now to Figure 12, the arm 40 indexes around its axis 48 in a clockwise motion until it enters the furnace 16. The furnace burns away the material from the used die held in portion 137 and the plug thus cleaning them.

The arm 40 indexes further in a clockwise motion until it registers with the loading ring 42, the loading cylinder 44 and the ejection cylinder 46. The now-cleaned die is brought into registration with the ejection cylinder 46 and an empty location on the loading ring 42. The ejection cylinder is activated driving the ejection piston upwards which displaces the die upwardly out of the transport arm 40, and into the empty location on the loading ring 42, where it is secured. The loading ring then indexes around to bring a new die which is required for a subsequent measurement operation. Once the new die registers with the transport arm 40, the loading cylinder 46 is activated driving the loading piston downwards which displaces the new die from the loading ring and pushes the new die into the transport arm 40.

The cleaned plug may be removed from the transport arm at any stage of the clockwise motion from the furnace 16 to the loading ring 42. A pick-up may come down and retrieve the cleaned plug from the loading ring to replace it on the cleaning piston 85. Alternatively the cleaned plug may be discarded at any stage before the next dirty plug enters the arm after

cleaning the the rheometer barrel. For example the cleaned plug could be ejected by one of the loading or ejection cylinders and collected. The clean plugs to be attached to the cleaning piston 85 may be from some other source.

Figure 14 shows a first prior art way of controlling a chemical reactor 210. The control system is referenced 212 and comprises a computer or microprocessor C, an input operational condition signal generator 216, and output signal communication means 218, 220 and 222. The reactor 210 comprises a feed hopper 224 communicating with a reactor chamber 226, heaters 228a, 228b and 228c provided along the elongate length of the reactor chamber 226, input feed stock material delivery means 230 delivering starting ingredients to the hopper 224, and a main drive motor 232 which provides the motive force to turn a feed screw (not shown) which mixes the feed stock and urges it along the elongate length of the reactor chamber 226.

The computer C receives signals indicative of the pressure, and in this example also of the temperature, in the reactor chamber 226. The output signal communication lines 218 to 222 lead to the motor 232, the feed stock delivery means 230, and the heaters 228a, 228b and 228c.

The computer controls the rate of feed of the feed stock via the delivery means, the speed of the drive motor 232, and hence the bulk flow rate of material passing through the reactor chamber 226, and the heaters 228a to 228c. The three heaters 228a to 228c can provide different temperature regimes in the flow path of the material in the reactor chamber.

The computer C has a read only memory (ROM) which contains a look-up chart, represented as a graph shown in Figure 15. This correlates pressure, as measured by sensor 216, and flow rate (derived from speed of the motor 232). This graph shows the relationship between the pressure and the flow rate which the computer sees as being ideal. The computer is programmed to try to maintain the ideal curve.

If the actual measured pressure T1 (as shown on Figure 15) for the flow rate (derived from the speed of the motor 232) lies on the curve, or falls within an allowable error band of the curve, then the computer decides not to alter any of its input variables (the temperature of heater 228a, heater 228b, heater 228c; the speed of the motor 232, measured delivery of the feed stock). If the point defined by the measured pressure, and the flow rate (either measured by a sensor, or calculated by the speed of the motor 232) lies off the curve, for example at point P2 in Figure 15, then the computer controls one or more of the input variables in a pre-programmed way which is designed to alter the conditions experienced in the reactor chamber 226 such as to tend to return the point P2 to the allowable curve.

It will be appreciated that the response time for the system is quite quick: of the order of one or two seconds. Simple physical conditions are measured and fed to the computer, which then refers them to a pre-recorded relationship to see whether they are allowable, or whether corrective action needs to be taken. This feedback control operates substantially instantly.

Figure 16 shows a second prior art control system. Similar components have been given the same reference numerals.

Instead of having a simple physical condition sensor, such as sensor 216 measuring the pressure and temperature, this system has an on-line rheometer 240 which is provided with a bleed line 242 which takes production material from the reactor 210, and its own independent drive motor 244. The motor 244 is in control of the computer, and the rheometer 240 provides signals indicative of the characteristics of the production material back to the computer C.

The on-line rheometer 240 drives the production material through a die, or more than one die, at a known pressure, by operation of the motor 244, and the flow rate of the material through the die is measured. This can provide information relating to the viscosity of the material. The temperature at which the measurements are made is kept constant. The rheometer 240 has its own heaters to achieve this, all of these are not shown.

The rheometer can take measurements of the viscosity of the material at different temperatures, under the control of the computer C, and also measurements of the viscosity of the material at different flow rates. Similarly, by having a variety of dies (possibly different dies of the same diameter but different lengths; or different dies of the same length but different diameters; or sets of different dies) different physical characteristics of the production material itself can be ascertained, and how they change with temperature/flow rate/pressure can be plotted. Thus the controller is fed information relating to the

physical characteristics of the material being produced in a far more detailed way than is the computer of Figure 14. It takes about 10 minutes for any one of the graphs 17a to 17d to be produced.

The computer controls the input parameters of the production system (motor speed, rate of delivery of starting materials, heater temperatures) in response to the signals from the rheometer 240 indicative of the physical characteristics of the material being produced. Figure 17a illustrates this.

In Figure 17a the computer C has had entered into it an ideal shear stress vs shear rate curve for the polymer that is being produced. This is referenced as 246. The results of a measurement run are shown as dotted line 248, and they are below the ideal curve. The computer adjusts one or more of the input parameters under its control so as to tend to move the measured curve back towards the ideal curve.

Unfortunately, because there is a 10 minute delay in the production of updated actual measured characteristics of the material there is a consequential delay in the control loop. This does not matter when the operator can be confident that there will be no sudden changes in the nature composition of the material being produced.

Figure 18 shows the new reactor system (comprising the reactor 210 and the control system 250). Again, components similar to those of Figures 14 or 16 have been given the same reference numerals.

An immediate pressure and temperature sensor 216 is also provided in this arrangement and sends signals

indicative of the pressure and temperature of a region of the reactor vessel 2 to the computer C. A second immediate physical condition sensor 252 is also provided, and this also provides signals indicative of the pressure and temperature of a second region. An on-line rheometer 240 is again provided.

By way of example, the on-line rheometer 240 may include a die such as that shown in Figure 21. This shows a die 254 having passages 256, 258, and 260 of three different lengths, but the same diameter. By passing production material through the passages at the same pressure (for example by applying the same body of production material to the upper face of the die) and measuring the relative flow rates we can determine information about the characteristics of the material being produced, for example its extensional viscosity.

The system shown in Figure 18 has in a random access memory (RAM) of the computer C a table/graph correlating the shear viscosity of the material being produced with a temperature at the point of measurement (this can be provided by rheometer 240). The viscosity of a plastics polymer is clearly of importance when it comes to deciding its chemical make up, and also its flow characteristics when it is used in a moulding process.

Figure 20 shows another table/graph held in a second RAM memory in the computer, and shows the relationship between pressure and flow rate. The pressure measured by sensor 216 is fed to the computer, which either knows the flow rate because it is controlling the motor 232, or actually measures it using the appropriate sensor. The pressure information, and flow rate information, are conveyed to the computer

practically continuously and instantaneously. The computer compares the measured point, for example point A1 on Figure 20 with an ideal curve, curve C1 which has been entered into the memory of the computer. If the point A1 lines on the curve C1 then no remedial action is required.

If the point A1 lies off the curve C1 by more than an allowable margin, for example as shown at point A2, then the computer takes action by controlling one or more of the variables under its control so as to tend to move the measured point back towards the desirable curve.

This is substantially the same as the arrangement of Figure 14.

However, the ideal curve C1 is updated periodically in response to information received from the on-line rheometer 240. This is quite different form the arrangement of Figure 14, and different from the arrangement of Figure 16.

The rheometer 240 periodically (for example every 10 minutes) provides a property one of production material: parameter two curve. In the example of Figure 19 this curve is the viscosity of the material compared with the temperature. The computer C also has in its memory an ideal graph for this curve, referenced curve C2 in Figure 19. If the measured curve falls upon curve C2, or is within an allowable margin of error, then the computer takes no action to alter curve C1. However, if the measure curve, referenced C3 in Figure 19, is too far away from the ideal curve C2 then the computer changes at least one of the input variables under its control in a way in which it is

designed to move the curve C3 closer to the curve C2. By so altering the input variables the computer will be moving one of the variables away from its ideal position on curve C1 (or its equivalent curve relating measured physical characteristic one to physical characteristic two). The computer modifies the entire curve C1 in response to finding that curve C3 is too far from ideal curve C2. The modified curve C1 is referenced as dotted curve C4 in Figure 20. This gives a new ideal curve - curve C4 the immediately measured simple physical conditions of the reactor. Thus the computer C then strives to maintain the conditions in the reactor such as to maintain new curve C4.

Thus the short-term control loop is modified by the long-term checking that the product being produced has the required physical characteristics. If it is found that, for whatever reason, the physical characteristics of the material being produced are not correct then the system automatically changes the short-term look-up tables/graphs in response.

An example of when the product being produced may change over time, even though the input variables are kept the same, might be if not all of the inhibitor or initiator of a reaction were used up during the flow process so that there was a gradual build up, which in time would influence the distribution of chain lengths of the polymer, and therefore influence the viscosity of the material being produced.

The present invention may enable the user to avoid having to key in values for a look-up table from which the computer will work. So long as the user is satisfied that the test means (rheometer 240) are showing that the syst m is producing a satisfactory

polymer he can initialise/normalise the input parameters of the short term graph of Figure 20 automatically. For example, the shape of the graph of Figure 20 will be the same, or roughly the same, for many polymers of slightly different composition. Thus if one point on the curve is known (because it is initialised to that) the system may automatically "draw in" the rest of the curve and use that as a basis for short term process control.

The system may be thought of as effectively automatically inputting its own look-up tables when the user indicates the desired input is being produced.

The system then modifies the short term responses to fluctuations in response to the long term check of the characteristics of the actual material introduced. If the curve on curve comparison shows a drift away from the ideal characteristic of the material being produced the computer either takes remedial action to get an acceptable curve-on-curve, and re-sets the immediately measurable conditions to the conditions required, or may sound an alarm. It may even shut the system down.

Instead of being self-initialising the system may require the original property one: parameter two look-up table/curve to be provided externally (either by keying it in or, as a plug in chip, or by data transfer, or by some other way). The system would then re-calibrate the immediately measure parameters required to produce the desired output polymer (by producing periodic parameter one: parameter two look-up tables/graphs and comparing them with the initialisation immediately measured variable graphs which it would either generate, or have provided to

it). If for any reason a new reaction condition parameter was required the system would re-set it to whatever value was necessary to achieve the desired end product curve, and thereafter operate a short-term control loop around that new parameter value.

CLAIMS

1. A chemical reactor apparatus comprising test means adapted in use to test a reaction product, or for testing an intermediate reaction product, reaction condition means adapted in use to detect one or more reaction conditions, and control means adapted in use to control at least one reaction input parameter, in which in use the reaction condition means provides to the control means a signal indicative of at least one reaction condition, and the test means provides the control means with a second signal indicative of a property of the reaction product being produced, and in which the control means utilises both the first and the second signals in controlling the reaction input parameter.

2. An apparatus according to claim 1, in which the control means comprises a long term loop used to modify the "target" curve for a short term control loop.

3. An apparatus according to claims 1 or 2, in which a reaction condition means produces a first signal indicative of the pressure at a region of the reactor apparatus.

4. An apparatus according to claims 1, 2 or 3, in which a reactor condition means produces a signal indicative of the temperature at a region of the reactor vessel.

5. An apparatus according to any preceding claim in which a reactor condition means produces a signal indicative of the bulk flow rate of material in the reactor vessel.

6. An apparatus according to any preceding claim in which a reactor condition means produces a signal indicative of the speed of drive of drive means for the reactor vessel.

7. An apparatus according to any preceding claim having reaction condition means indicative of the amount of, or ratio between, first and second start materials being input to the reactor vessel.

8. An apparatus according to any preceding claim, in which the reaction condition means comprises a single sensor.

9. An apparatus according to any one of claims 1 to 8, in which the reaction condition means comprises a plurality of sensors which detect different physical conditions.

10. An apparatus according to any one of claims 1 to 8, in which the reaction condition means are provided with more than one sensor to detect the same physical condition.

11. An apparatus according to claims 9 or 10, in which the sensors are at different locations.

12. An apparatus according to any preceding claim, in which the test means provide signals indicative of the shear viscosity of the material being tested.

13. An apparatus according to any preceding claim, in which the test means provide signals indicative of the die swell of the material.

14. An apparatus according to any preceding claim, in which the test means provide signals indicative of the extensional viscosity of the material.

15. An apparatus according to any preceding claim, in which the test means provide signals indicative of the wallslip of the material.

16. An apparatus according to any preceding claim, in which the test means provide signals indicative of a pair of the characteristics of the material, or any combination of the characteristics.

17. An apparatus according to any preceding claim, in which the test means is provided with more than one sensor.

18. An apparatus according to any preceding claim, in which the test means comprises a die.

19. An apparatus according to claim 18, in which the die has a plurality of holes of different sizes.

20. An apparatus according to any preceding claim, in which the control means responds to signals from the reaction condition means as a short term control of the reaction conditions.

21. An apparatus according to any preceding claim, in which the control means responds to signals from the test means as a long term check on the characteristics of what is being produced.

22. An apparatus according to any preceding claim, in which signals from the test means are used by the control means to amend or alter a reaction condition

look-up table or parameter one: parameter two graph held in a memory of the control means, said reaction condition graph being used by the control means to control said reaction input parameter in response to signals from said reaction condition means.

23. An apparatus according to claim 22, having a parameter one: parameter two look-up table/graph held in RAM, and means for altering the table/graph in response to signals indicative of the properties of material being tested.

24. An apparatus according to any preceding claim, in which the control means has a memory to which is written signals from the test means, or the reactor condition means, or both.

25. An apparatus according to claim 24, in which the reaction product is adapted to be produced and checked by the test means, the input parameters required to achieve a deserved result being written to the memory.

26. An apparatus according to any preceding claim, in which the control means checks or updates its reaction condition parameters periodically.

27. An apparatus according to claim 26, in which the control means monitors or controls its input reaction parameter at about 1 second, 2 second, or 3 second intervals.

28. An apparatus according to claims 26 or 27, in which the control means monitors and/or corrects its input reaction parameter at intervals of a first time period, and updates or monitors a 1 ok-up table/graph

that it uses to monitor its input reaction parameter at a second, longer, interval.

29. A method of controlling a production reactor adapted to produce a production material from one or more starting materials, the method comprising the steps of having a look-up table, graph or other concordance between a first physical condition measured in the reactor or input to the reactor and a second physical condition measured in the reactor or input to the reactor; using the look-up table or other concordance to control an input parameter of the reactor so as to try to maintain stable the nature of the production material; testing one or more physical characteristics of the production material; and using the results of the testing of the physical characteristics of the material to modify the look-up table or other concordance, or to modify how that table or concordance is used in the control of the input parameter.

30. A control system for reaction apparatus, the control system being adapted to enable the reaction apparatus to be in accordance with any preceding claim.

31. A control system for a reactor having test means for testing characteristics of the material being produced by the reactor, and also having condition sensing means for detecting conditions being experienced in the reactor; signals from the test means and from the condition sensing means both being used in the control of the reactor.

32. A reactor system having an in-line sensor in which material is moved past the sensor by the reactor's

drive means, as opposed to any independently controlled drive means.

33. A rheometer comprising a die, a material holding chamber and a cleaning means; in which the cleaning means is adapted to clean the material holding chamber automatically.

34. A rheometer according to claim 33 in which the cleaning means comprises a plug and a means for urging the plug through the material holding chamber.

35. A rheometer according to claim 34 in which the plug is cylindrical.

36. A rheometer according to claim 34 or claim 35 in which the plug has a generally uniform outer surface.

37. A rheometer according to any of claims 34 to 36 in which the plug has a stepped surface having at least one raised region adjacent at least one sunken region.

38. A rheometer according to claim 37 in which the raised regions and the sunken regions on the plug are rings.

39. A rheometer according to claim 38 in which the plug has the form of a series of rings of alternating wider and narrower diameter.

40. A rheometer according to any of claims 34 to 39 in which the plug is provided in delivery means adapted to deliver the plug to the entrance to the rheometer bore.

41. A rheometer according to claim 40 in which the delivery means comprises a plug holding bore in a body.

42. A rheometer according to any of claims 34 to 41 in which the plug is adapted to change diameter when inserted into the material handling chamber.

43. A rheometer according to any of claims 34 to 42 in which the plug is resilient.

44. A rheometer according to claim 42 in which the plug is compressed from a wider diameter to a narrower diameter when inserted into the material handling chamber.

45. A rheometer according to any one of claims 34 to 44 in which the plug is adapted to expand in the material handling chamber.

46. A rheometer according to any of claims 42, 44 or 45 in which change in diameter of the plug is achieved by thermal expansion.

47. A rheometer according to any of claims 34 to 46 in which the plug is a complementary shape to the material holding chamber so as to be a tight interference sliding fit in it.

48. A rheometer according to any of claims 34 to 46 in which the plug is adapted to thermally expand in the material holding chamber such that the outer surface of the plug is in bearing contact with the inner surface of the material holding chamber.

49. A rheometer according to any of claims 34 to 48 in which comprises a body portion and a split ring surrounding a region of the body portion.

50. A rheometer according to any of claims 34 to 49 in which the plug, or elements of the plug, adapted to be in contact with the material handling space are made of phosphor bronze.

51. A rheometer according to any of claims 34 to 49 in which the plug, is made of wood.

52. A rheometer according to any of claims 34 to 49 in which the plug of polymeric material.

53. A rheometer according to claim 52 in which the plug is made of "Tufnol".

54. A rheometer according to any of claims 34 to 53 in which the plug comprises an elongate body having one or more split rings mounted on it.

55. A rheometer according to any of claims 34 to 54 in which the plug is re-usable.

56. A rheometer according to any of claims 34 to 54 in which the plug is disposable.

57. A rheometer according to any of claims 34 to 56 in which the plug is a spring.

58. A rheometer according to claim 57 in which the plug is a helical spring adapted to bear against the inner surface of the material handling chamber when located therein.

59. A rheometer according to claim 58 in which the length of the spring is reduced in order to achieve radial expansion.

60. A rheometer according to any of claims 34 to 59 in which the plug is provided additionally with flexible cleaning material.

61. A rheometer according to any one of claims 33 to 60 in which the material holding chamber is an elongate bore.

62. A method of operating a rheometer comprising the steps of pushing melt from a material holding space through a die with motive means, removing the motive means from the material holding space, cleaning the motive means, and refilling the material holding chamber; in which the operations of refilling of the holding space and cleaning of the motive means overlap in the same period in time.

63. A method of operating a rheometer according to claim 62 in which the cleaning operation and refilling operation are automatic.

64. A rheometer comprising a material holding space, die means, tamping means, and motive means adapted to push material from the material holding space through the die; in which the tamping means and the motive means are different components.

65. A rheometer according to any one of claims 33 to 64 which comprises a material holding chamber, a die, tamping means, and motive means adapted to push material from the material holding chamber through the die; in which the tamping means and the motive means are different components.

66. A rheometer according to claim 64, or claim 65 in which the motive means and the tamping means are both elongate.

67. A rheometer according to any of claims 64 to 66 in which the motive means and the tamping means extend in the same direction.

68. A rheometer according to any of claims 64 to 67 in which the motive means and the tamping means comprise elongate rods.

69. A rheometer according to any of claims 64 to 68 in which the motive means and the tamping means may be moved simultaneously.

70. A rheometer according to any of claims 64 to 69 in which the tamping means and the motive means are both mounted on the same mounting member.

71. A rheometer according to claim 70 in which the mounting member is swivelable.

72. A rheometer according to claim 70 to claim 71 in which the mounting member is adapted to index from a first position to a second position.

73. A rheometer according to claim 72 in which in the second position the motive means is at a cleaning station to be cleaned.

74. A rheometer according to any of claims 64 to 73 in which the motive means is a piston.

75. A rheometer according to any one of claims 33 to 74 which comprises a die and a material holding

chamber; in which heating means is provided adjacent the die.

76. A rheometer comprising a die means and a material holding space; in which heating means is provided adjacent the die.

77. A rheometer according to claim 75 or claim 76 in which the heating means is provided below the die.

78. A rheometer according to any of claims 75 to 78 in which the material holding space is a rheometer barrel.

79. A rheometer according to any of claims 75 to 78 in which the heating means comprises one or more heaters.

80. A rheometer according to any of claims 75 to 79 in which the heating means below the die is controlled by a controller which receives temperature signals.

81. A method of filling a material holding space in a rheometer comprising the steps of loading the material holding space with a quantity of material, tamping the material, and loading the material holding space with a further quantity of material.

82. A method according to claim 81 in which a series of tamping operations are performed.

83. A method according to claim 81 or claim 82 in which a loading means is used to load the material holding space.

84. A method according to claim 83 in which the loading means comprises a carousel.

85. A rheometer according to any one of claims 33 to 84 which comprises a die, a material holding chamber, tamping means, sensor means adapted to measure a parameter of the rheometer, and control means adapted to control the operation of the tamping means in response to the measurement of the parameter.

86. A rheometer comprising a die, a material holding space, tamping means, sensor means adapted to measure a parameter of the rheometer, and control means adapted to control the operation of the tamping means in response to the measurement of the parameter.

87. A rheometer according to claim 85 or claim 86 in which the control means changes the operation of the tamping means.

88. A rheometer according to any of claims 85 to 87 in which heating occurs during the tamping operation.

89. A rheometer according to any one of claims 33 to 88 with die transfer means which is adapted to collect a die when it is moved from its operative position in the rheometer, and transfer the die to a cleaning station.

90. A rheometer with die transfer means which is adapted to collect a die when it is moved from its operative position in the rheometer, and transfer the die to a cleaning station.

91. A rheometer according to claims 89 or 90 in which collection means are provided to collect a die from a rheometer.

92. A rheometer according to any of claims 89 to 90 in which the cleaning station is a furnace.

93. A rheometer according to any one of claims 33 to 92 having automatic die changing means.

94. A rheometer having automatic die changing means.

95. A rheometer according to claim 93 or claim 94 in which the automatic die changing means has a plurality of interchangeable dies of different characteristics.

96. A rheometer according to any of claims 93 to 96 in which the automatic die changing means comprises a cartridge or block having a plurality of dies and a corresponding plurality of operative positions with respect to a flow passageway of the rheometer with which the operative die is in communication.

97. A rheometer according to any of claims 93 to 96 in which cleaning plug holding means are provided in the cartridge.

98. A rheometer according to any of claims 93 to 97 in which seal means to seal the die that is in use to the flow passageway are provided.

99. A rheometer according to any one of claims 33 to 98 comprising a die, a material holding chamber and cooling means; in which the cooling means is adapted to cool the material which emerges from the die.

100. A rheometer comprising a die, a material holding space and cooling means; in which the cooling means is adapted to cool the material which emerges from the die.

101. A rheometer according to claim 99, or claim 100 in which the cooling means is one or more air jets.

102. A rheometer according to any one of claims 33 to 101 comprising a die, a material holding chamber, and cutting means; in which the cutting means is adapted to cut the material which emerges from the die when the extrudate reaches a pre-determined length.

103. A rheometer comprising a die, a material holding space, and cutting means; in which the cutting means is adapted to cut the material which emerges from the die when the extrudate reaches a pre-determined length.

104. A rheometer according to claim 102 or claim 103 in which sensor means are provided to measure the length of the extruded material.

105. A rheometer according to any of claims 102 to 104 in which diameter measuring means is provided.

106. A rheometer according to any of claims 102 to 105 in which a parameter indicative of swell is measured.

107. A rheometer according to any one of claims 33 to 106 comprising a die, a material holding chamber, and cutting means; in which the cutting means is adapted to hold the extruded material against lateral movement during cutting of the extruded material.

108. A rheometer comprising a die, a material holding space, and cutting means; in which the cutting means is adapted to hold the extruded material against lateral movement during cutting of the extruded material.

109. A rheometer according to claim 107 or claim 108 in which the cutting means are pincer cutters.

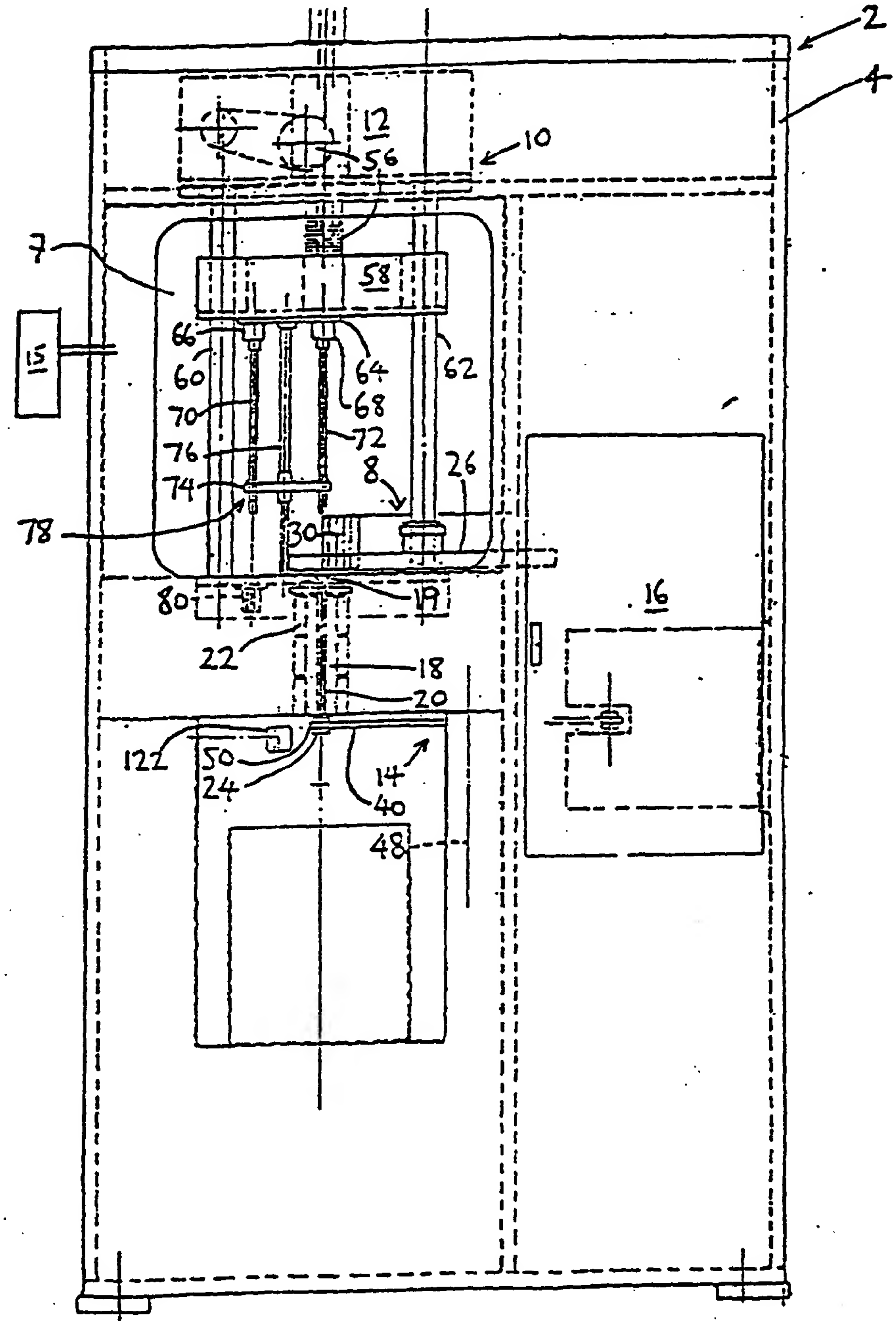

2
4
12
10
56
7
58
15
66
60
64
62
68
70
72
76
8
26
74
78
30
80
19
22
18
20
122
50
24
14
40
48
16

FIG.1

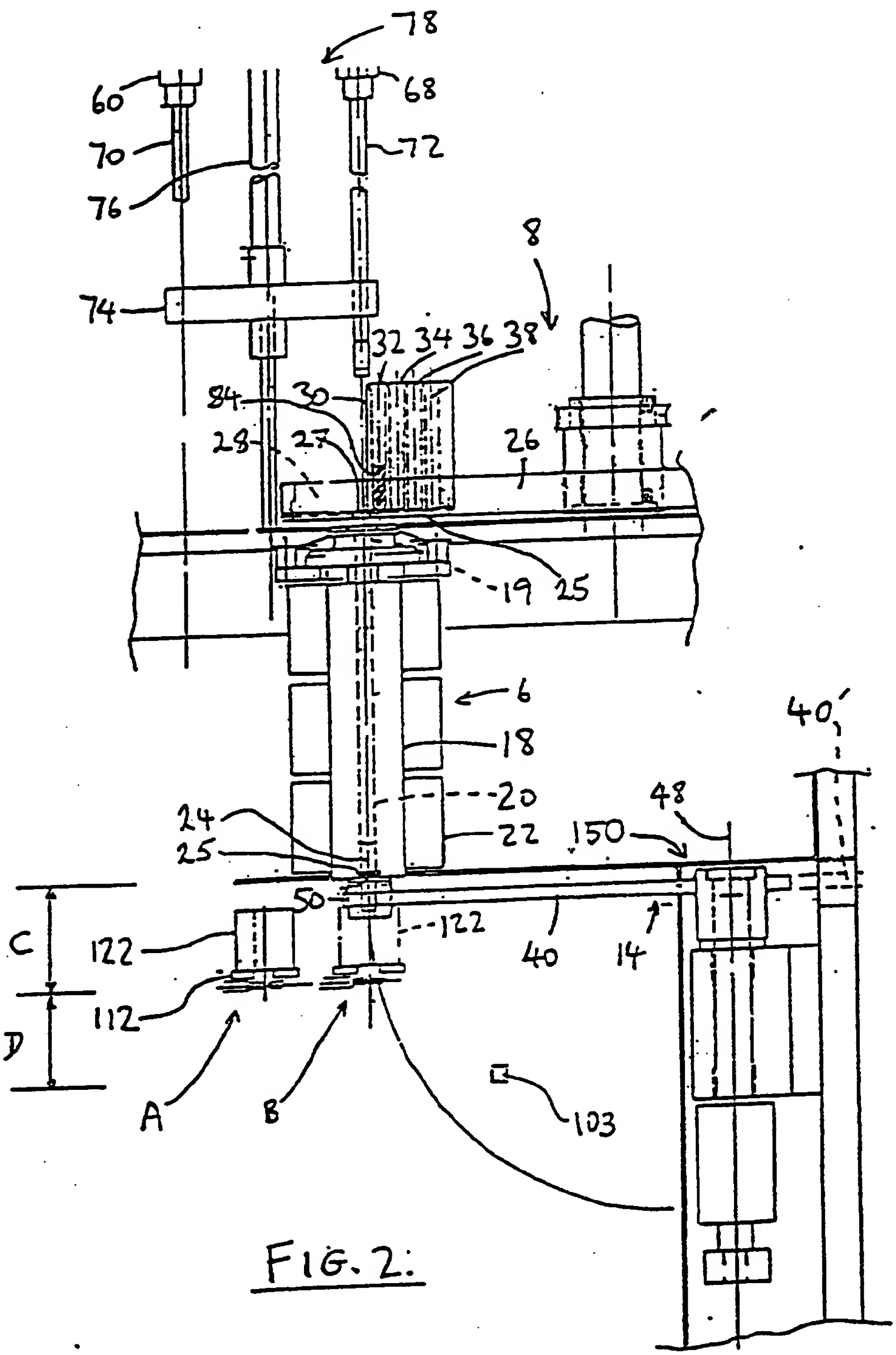

78
60
68
70
72
76
74
8
32 34 36 38
84
30
28
27
26
19
25
6
18
20
22
24
25
40'
48
150
50
122
40
14
C
122
112
D
A
B
103

FIG. 2.

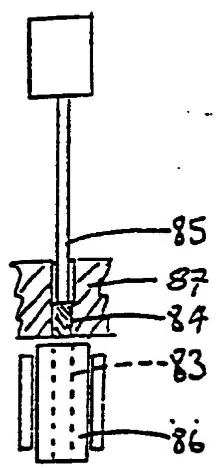


FIG. 3.

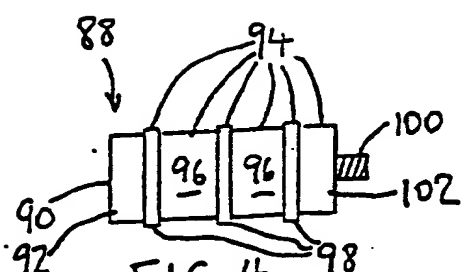


FIG. 4.

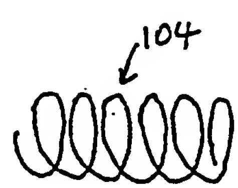


FIG. 5.

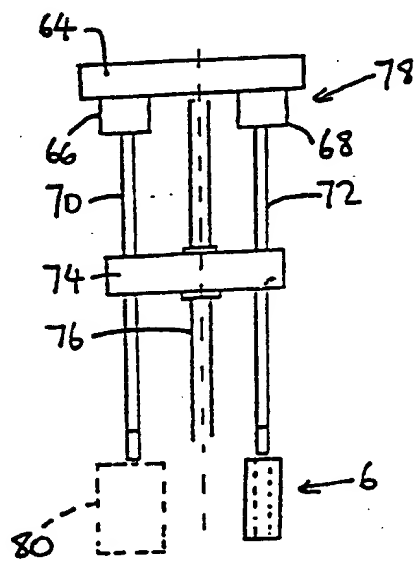


FIG. 7.

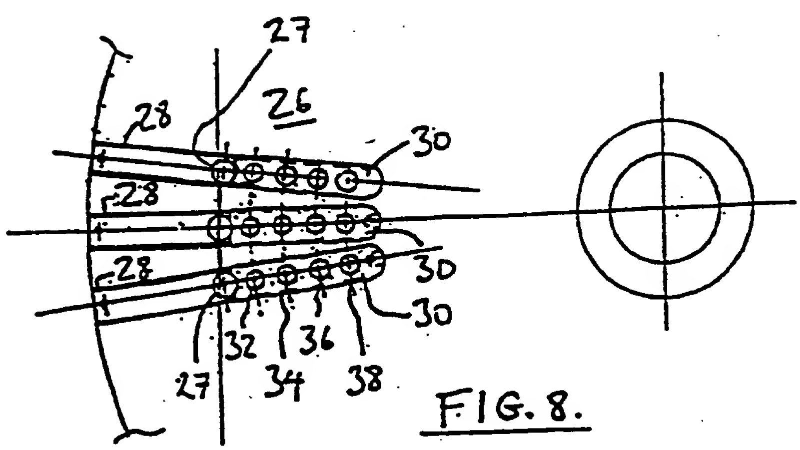


FIG. 8.

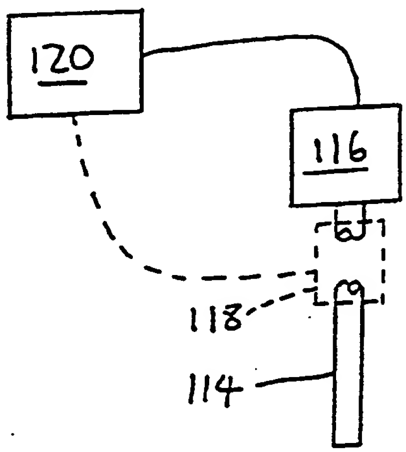


FIG. 9.

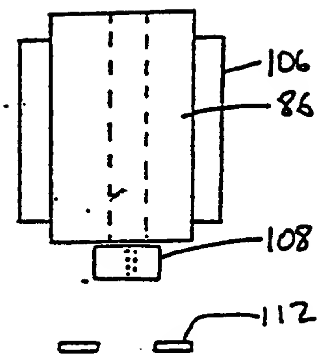


FIG. 10.

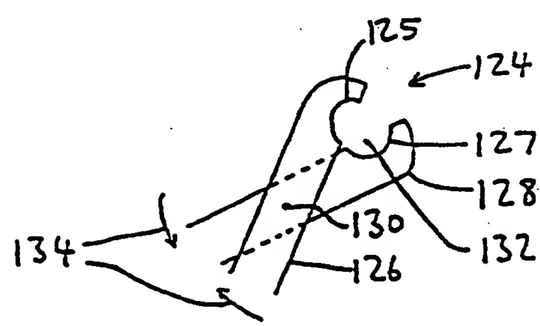


FIG. 11.

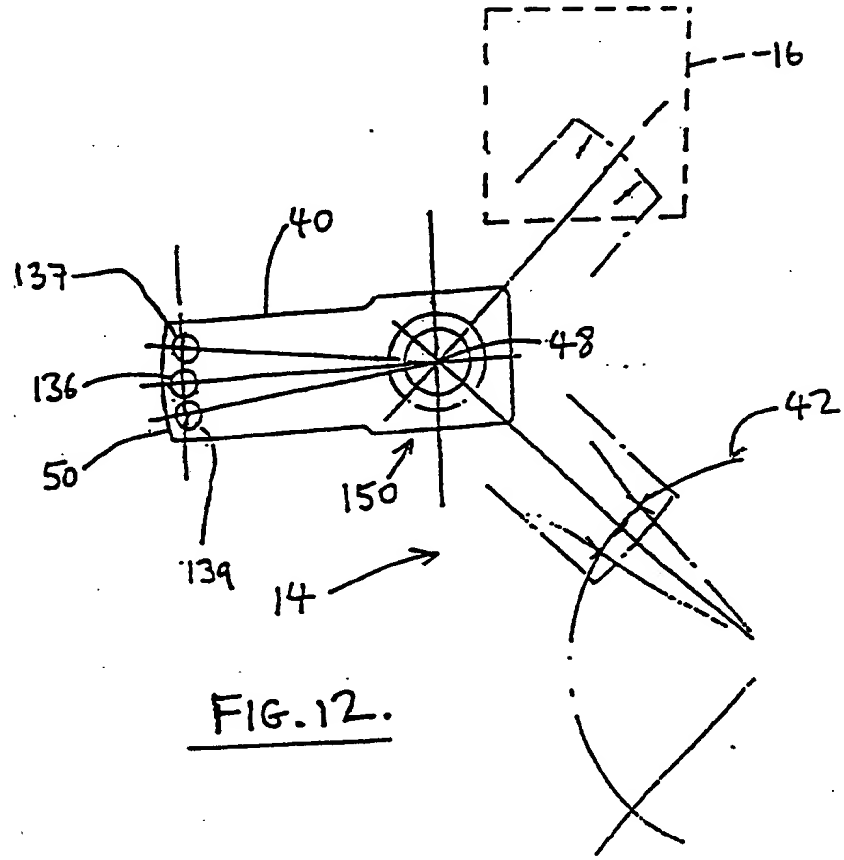


FIG. 12.

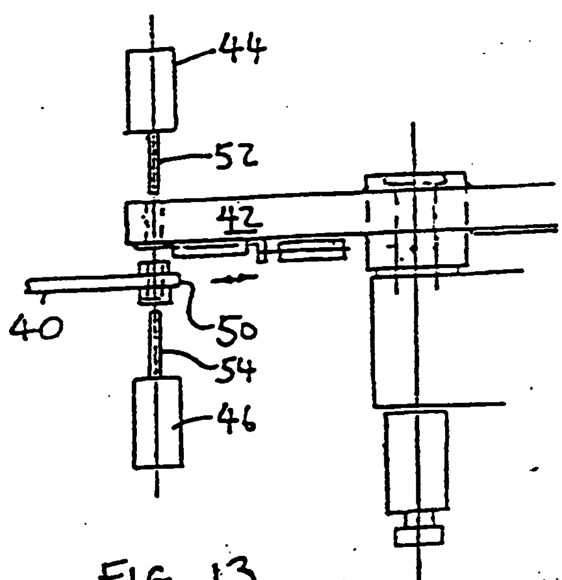


FIG. 13.

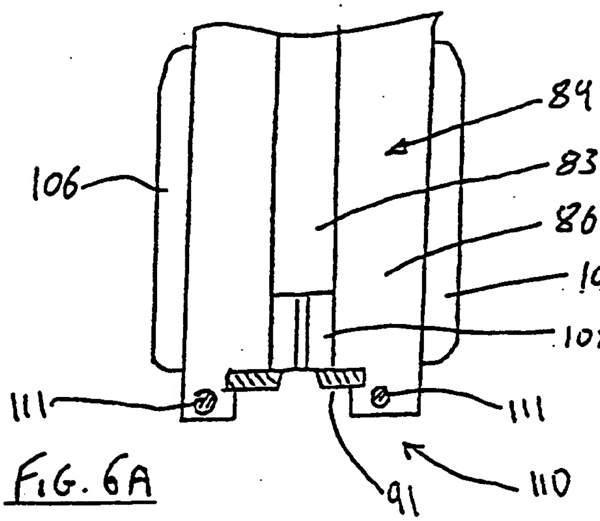


FIG. 6A

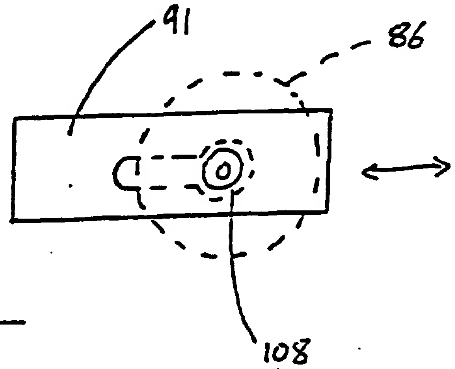
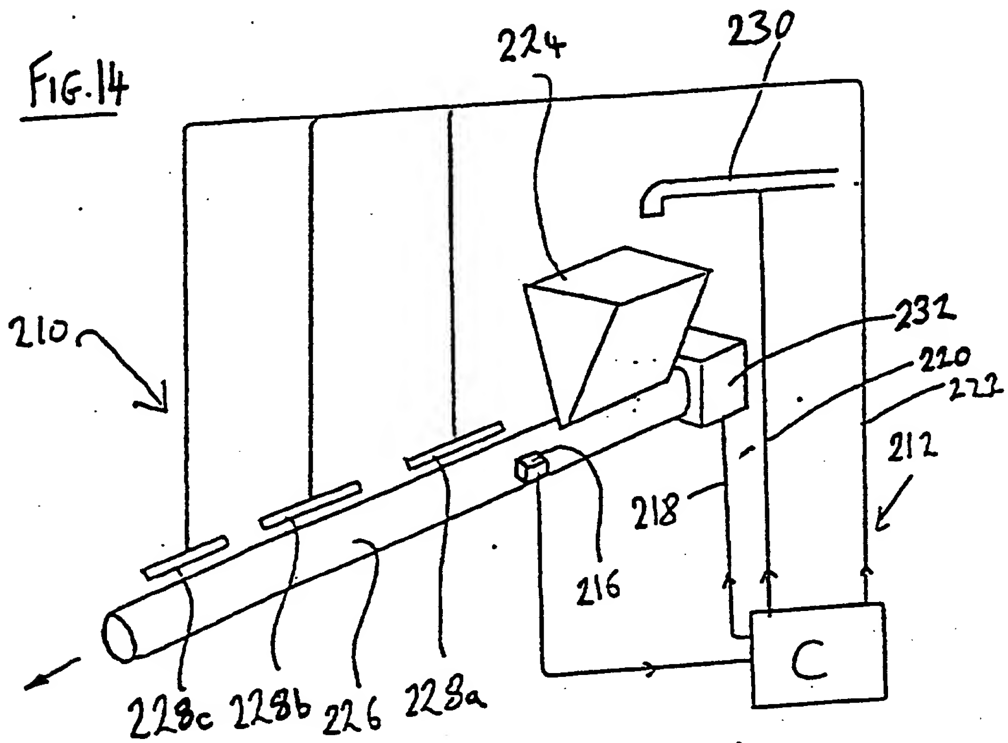


FIG. 6B

FIG.14


224
230
210
232
220
222
212
218
216
C
228c
228b
226
228a

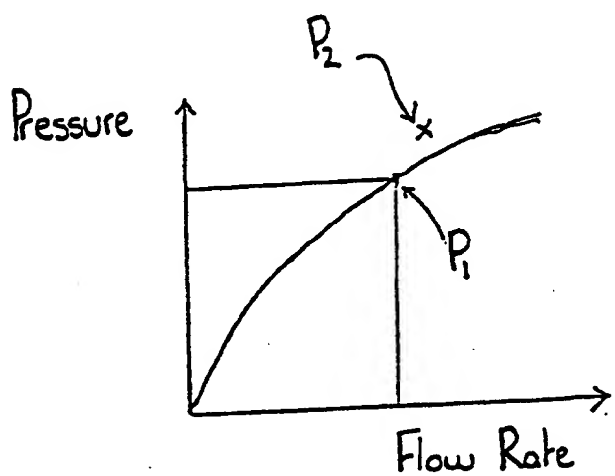

P_2
Pressure
P_1
Flow Rate

FIG.15

FIG. 16

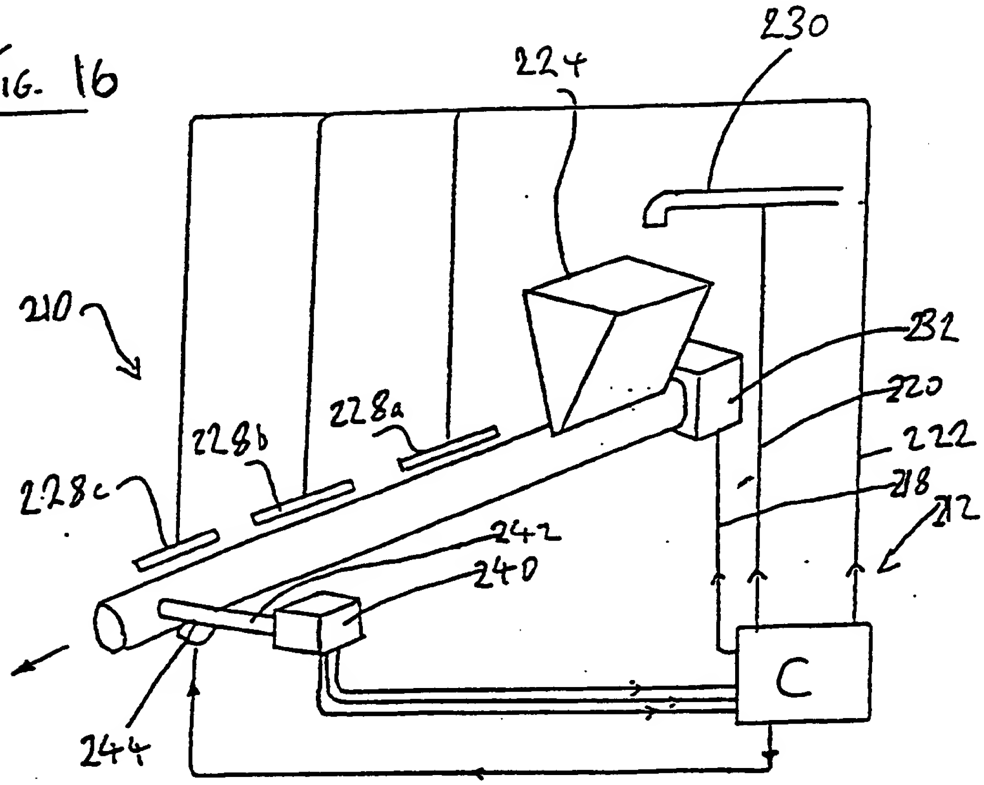


FIG. 17A

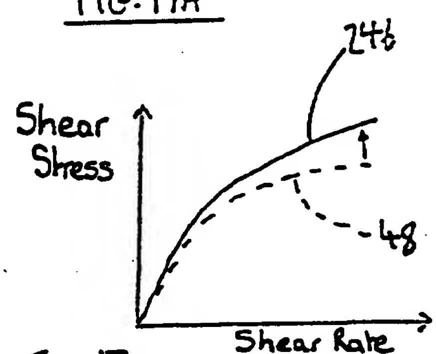


FIG. 17B

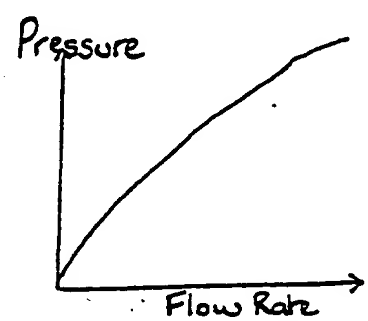


FIG. 17C

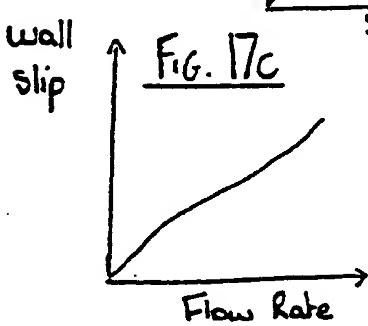


FIG. 17D

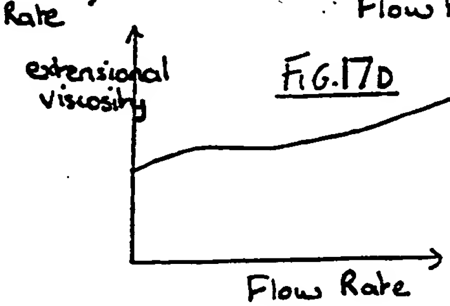


FIG. 19

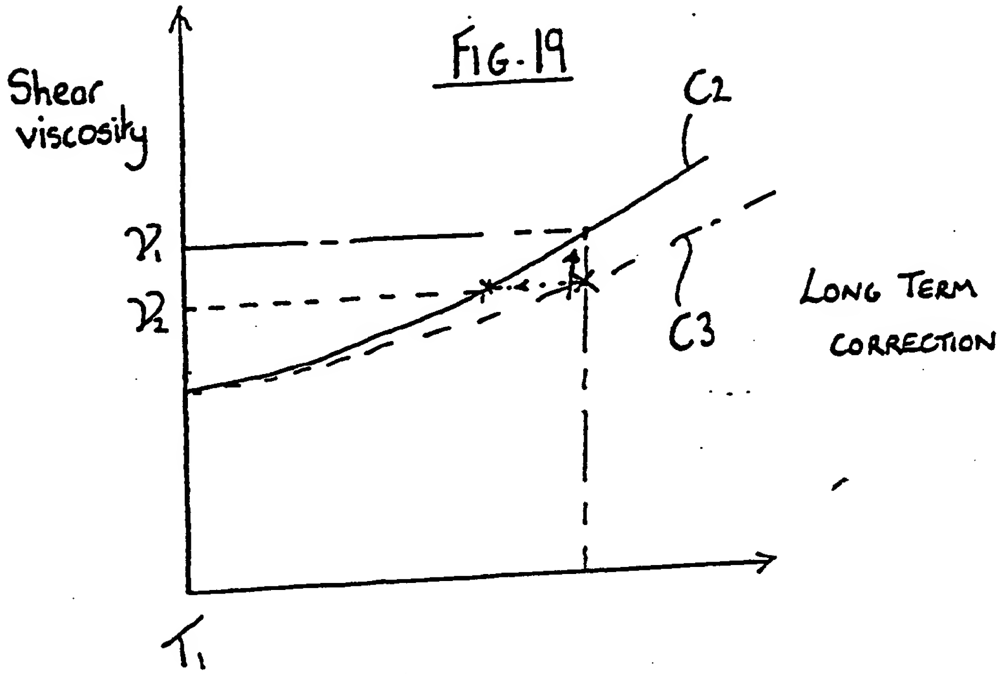

Shear viscosity
C2
ν_1
ν_2
C3
LONG TERM CORRECTION
T_1

FIG. 20

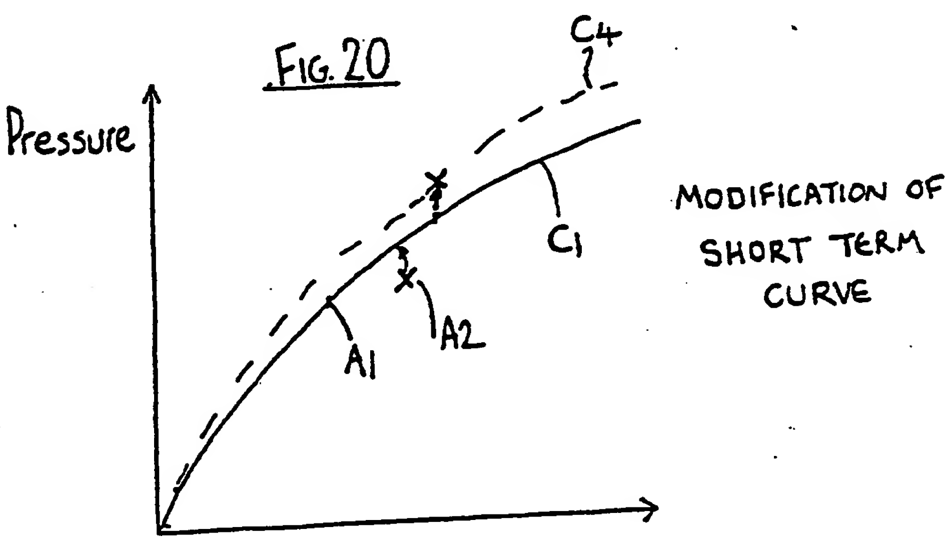
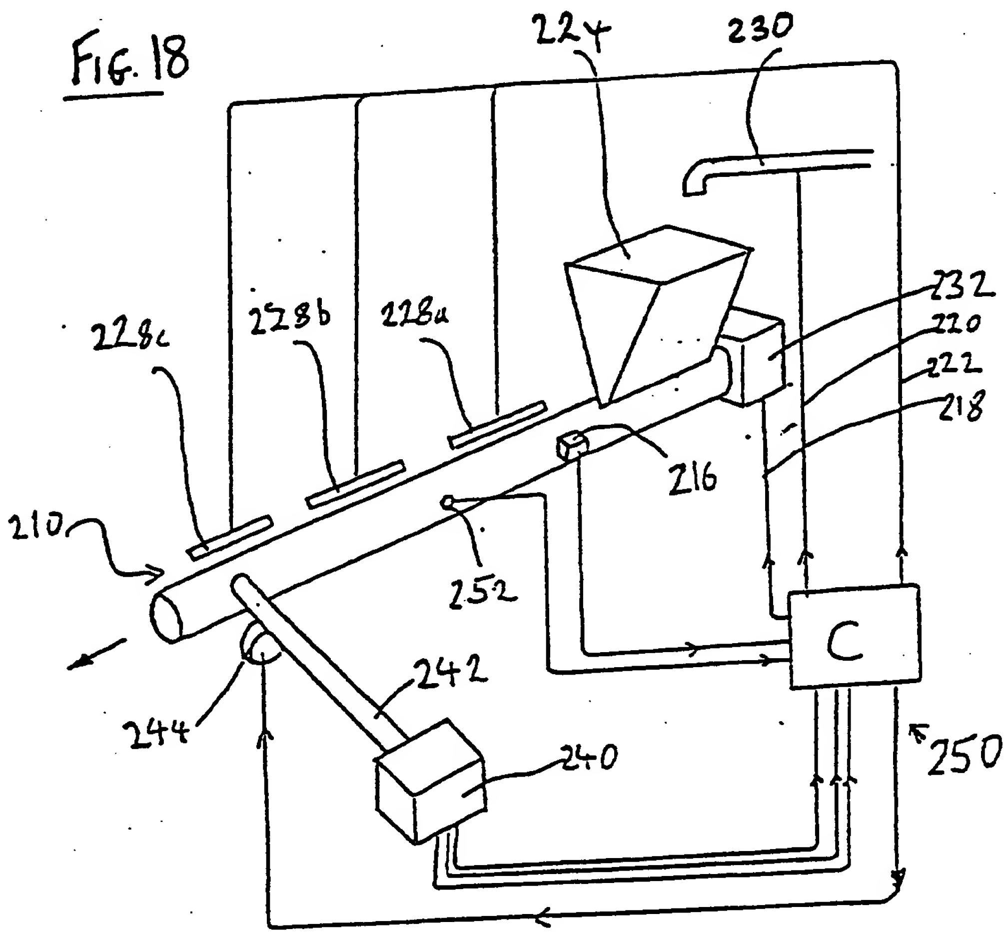

C4
Pressure
C1
A1
A2
MODIFICATION OF SHORT TERM CURVE

Fig. 18


224
230
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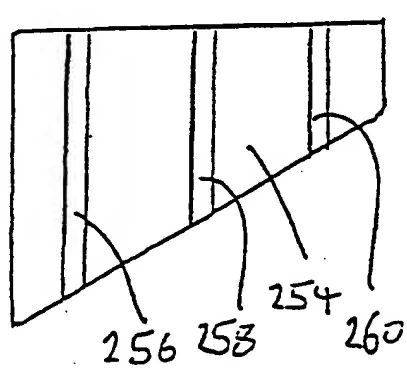
220
222
218
228c
228b
228a
216
210
252
C
242
244
240
250

Fig. 21


256
258
254
260